

**What is Claimed is:**

- 1           1.       A process for the manufacture of a  $\text{LiMPO}_4$  powder, comprising the steps  
2 of:  
3                   providing an equimolar aqueous solution of  $\text{Li}^{1+}$ ,  $\text{M}^{n+}$ , and  $\text{PO}_4^{3-}$  prepared  
4 by dissolving components which are susceptible to coexist as solutes in an aqueous  
5 system and which, upon heating at a temperature below  $500^\circ\text{C}$ , decompose to form a  
6 pure homogeneous Li and M phosphate precursor;  
7                   evaporating water from the solution, thereby producing a solid mixture;  
8                   decomposing the solid mixture at a temperature below  $500^\circ\text{C}$  to form a  
9 pure homogeneous Li and M phosphate precursor; and  
10                  annealing the precursor at a temperature of less than  $800^\circ\text{C}$  in an inert or  
11 reducing atmosphere, thereby forming a  $\text{LiMPO}_4$  powder;  
12                  wherein  $\text{M}^{n+}$  is one or more of  $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Co}^{2+}$ ,  $\text{Ni}^{2+}$ , and  $\text{Mn}^{2+}$ , and M is  
13  $\text{Fe}_x\text{Co}_y\text{Ni}_z\text{Mn}_w$ , with  $0 \leq x \leq 1$ ,  $0 \leq y \leq 1$ ,  $0 \leq z \leq 1$ ,  $0 \leq w \leq 1$ , and  $x + y + z + w = 1$ .
- 1           2.       The process according to claim 1, wherein in the step of annealing the  
2 precursor, the annealing temperature is less than  $600^\circ\text{C}$ .
- 1           3.       A process for the manufacture of a  $\text{LiFePO}_4$  powder, comprising the  
2 steps of:  
3                   providing an equimolar aqueous solution of  $\text{Li}^{1+}$ ,  $\text{Fe}^{3+}$ , and  $\text{PO}_4^{3-}$  prepared  
4 by dissolving components which are susceptible to coexist as solutes in an aqueous  
5 system and which, upon heating at a temperature below  $500^\circ\text{C}$ , decompose to form a  
6 pure homogeneous Li and Fe phosphate precursor;  
7                   evaporating water from the solution, thereby producing a solid mixture;  
8                   decomposing the solid mixture at a temperature below  $500^\circ\text{C}$  to form a  
9 pure homogeneous Li and Fe phosphate precursor; and  
10                  annealing the precursor at a temperature of less than  $800^\circ\text{C}$  in a reducing  
11 atmosphere, thereby forming a  $\text{LiFePO}_4$  powder.
- 1           4.       The process according to claim 3, wherein in the step of annealing the  
2 precursor, the annealing temperature is less than  $600^\circ\text{C}$ .
- 1           5.       The process according to claims 3, wherein the  $\text{Fe}^{3+}$  bearing component  
2 is iron nitrate.

1           6.       A powder for use in lithium insertion-type electrodes with a formula  
2        $\text{LiMPO}_4$  having an average particle size of less than  $1\mu\text{m}$ , wherein M is  $\text{Fe}_x\text{Co}_y\text{Ni}_z\text{Mn}_w$ ,  
3       with  $0 \leq x \leq 1$ ,  $0 \leq y \leq 1$ ,  $0 \leq z \leq 1$ ,  $0 \leq w \leq 1$ ,  $x + z + w > 0$ , and  $x + y + z + w = 1$ .

1           7.       The powder according to claim 6, wherein M is Fe, the powder having a  
2       reversible electrode capacity of at least 65% of a theoretical capacity when used as an  
3       active component in a cathode that is cycled between 2.70 and 4.15 V vs.  $\text{Li}^+/\text{Li}$  at a  
4       discharge rate of C/5 at  $25^\circ\text{C}$ .

1           8.       A powder for use in lithium insertion-type electrodes prepared by a  
2       process comprising the steps of:  
3                    providing an equimolar aqueous solution of  $\text{Li}^{1+}$ ,  $\text{M}^{n+}$ , and  $\text{PO}_4^{3-}$  prepared  
4       by dissolving components which are susceptible to coexist as solutes in an aqueous  
5       system and which, upon heating at a temperature below  $500^\circ\text{C}$ , decompose to form a  
6       pure homogeneous Li and M phosphate precursor;  
7                    evaporating water from the solution, thereby producing a solid mixture;  
8                    decomposing the solid mixture at a temperature below  $500^\circ\text{C}$  to form a  
9       pure homogeneous Li and M phosphate precursor; and  
10                  annealing the precursor at a temperature of less than  $600^\circ\text{C}$  in an inert or  
11       reducing atmosphere, thereby forming a  $\text{LiMPO}_4$  powder;  
12                   wherein  $\text{M}^{n+}$  is one or more of  $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Co}^{2+}$ ,  $\text{Ni}^{2+}$ , and  $\text{Mn}^{2+}$ , and M is  
13        $\text{Fe}_x\text{Co}_y\text{Ni}_z\text{Mn}_w$ , with  $0 \leq x \leq 1$ ,  $0 \leq y \leq 1$ ,  $0 \leq z \leq 1$ ,  $0 \leq w \leq 1$ , and  $x + y + z + w = 1$ .

1           9.       The powder according to claim 8, wherein  $\text{M}^{n+}$  is  $\text{Fe}^{3+}$ , M is Fe, and the  
2       annealing occurs in a reducing atmosphere.

1           10.      A battery comprising a lithium insertion-type electrode including a  
2       powder prepared by a process comprising the steps of:  
3                    providing an equimolar aqueous solution of  $\text{Li}^{1+}$ ,  $\text{M}^{n+}$ , and  $\text{PO}_4^{3-}$  prepared  
4       by dissolving components which are susceptible to coexist as solutes in an aqueous  
5       system and which, upon heating at a temperature below  $500^\circ\text{C}$ , decompose to form a  
6       pure homogeneous Li and M phosphate precursor;  
7                    evaporating water from the solution, thereby producing a solid mixture;  
8                    decomposing the solid mixture at a temperature below  $500^\circ\text{C}$  to form a  
9       pure homogeneous Li and M phosphate precursor; and

10                    annealing the precursor at a temperature of less than 600° C in an inert or  
 11                    reducing atmosphere, thereby forming a  $\text{LiMPO}_4$  powder;  
 12                    wherein  $\text{M}^{n+}$  is one or more of  $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Co}^{2+}$ ,  $\text{Ni}^{2+}$ , and  $\text{Mn}^{2+}$ , and M is  
 13                     $\text{Fe}_x\text{Co}_y\text{Ni}_z\text{Mn}_w$ , with  $0 \leq x \leq 1$ ,  $0 \leq y \leq 1$ ,  $0 \leq z \leq 1$ ,  $0 \leq w \leq 1$ , and  $x + y + z + w = 1$ .

1                    11.        The battery according to claim 10, wherein the powder has an average  
 2                    particle size of less than  $1\mu\text{m}$  and  $x + z + w > 0$ .

1                    12.        The battery according to claim 11, wherein M is Fe, the powder having a  
 2                    reversible electrode capacity of at least 65% of a theoretical capacity when used as an  
 3                    active component in a cathode that is cycled between 2.70 and 4.15 V vs.  $\text{Li}^+/\text{Li}$  at a  
 4                    discharge rate of C/5 at 25° C.

1                    13.        The battery according to claim 10, wherein  $\text{M}^{n+}$  is  $\text{Fe}^{3+}$ , M is Fe, and the  
 2                    annealing occurs in a reducing atmosphere.

1                    14.        A process for the manufacture of a lithium insertion-type electrode  
 2                    comprising the steps of:  
 3                                       providing an equimolar aqueous solution of  $\text{Li}^{1+}$ ,  $\text{M}^{n+}$ , and  $\text{PO}_4^{3-}$  prepared  
 4                    by dissolving components which are susceptible to coexist as solutes in an aqueous  
 5                    system and which, upon heating at a temperature below 500° C, decompose to form a  
 6                    pure homogeneous Li and M phosphate precursor;  
 7                                       evaporating water from the solution, thereby producing a solid mixture;  
 8                                       decomposing the solid mixture at a temperature below 500° C to form a  
 9                    pure homogeneous Li and M phosphate precursor;  
 10                                       annealing the precursor at a temperature of less than 600° C in an inert or  
 11                    reducing atmosphere, thereby forming a  $\text{LiMPO}_4$  powder;  
 12                                       providing a mixture of the  $\text{LiMPO}_4$  powder and a conductive carbon  
 13                    bearing powder; and  
 14                                       milling the mixture during a period of time to optimize a reversible  
 15                    electrode capacity of the electrode;  
 16                                       wherein  $\text{M}^{n+}$  is one or more of  $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Co}^{2+}$ ,  $\text{Ni}^{2+}$ , and  $\text{Mn}^{2+}$ , and M is  
 17                     $\text{Fe}_x\text{Co}_y\text{Ni}_z\text{Mn}_w$ , with  $0 \leq x \leq 1$ ,  $0 \leq y \leq 1$ ,  $0 \leq z \leq 1$ ,  $0 \leq w \leq 1$ , and  $x + y + z + w = 1$ .

1           15.     The process according to claim 14, wherein M is Fe, the conductive  
2 carbon powder is one of Acetylene Black and Carbon Super P, the weight ratio of  
3  $\text{LiFePO}_4$ /carbon is between 75/25 and 85/15, and the milling time is between 15 and 25  
4 minutes.

1           16.     The process according to claim 14, wherein the powder has an average  
2 particle size of less than  $1\mu\text{m}$  and  $x + z + w > 0$ .

1           17.     The process according to claim 16, wherein M is Fe and the reversible  
2 electrode capacity is at least 65% of a theoretical capacity when used as an active  
3 component in a cathode that is cycled between 2.70 and 4.15 V vs.  $\text{Li}^+/\text{Li}$  at a discharge  
4 rate of C/5 at  $25^\circ\text{C}$ .